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Sir:

Transmitted herewith for filing is the patent application of:

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FOR: VALVE FOR LIQUID SEPARATION

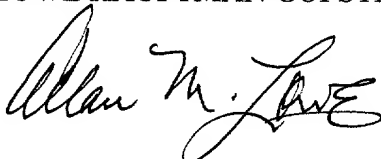
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Enclosed are:

- [X] 10 pages of specification, claims, abstract
- [] Declaration & Power of Attorney
- [X] Priority Claimed
- [X] Certified copy of German Patent Application No. 199 46 654.8
- [X] An assignment of the invention to and the assignment recordation fee
- [X] Return Receipt Postcard
- [X] Information Disclosure Statement, Form PTO-1449
- [X] Copies of IDS Citations
- [X] Preliminary Amendment

Respectfully submitted,

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Valve for liquid separation

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The invention concerns a valve for liquid separation, especially for analytical or preparative high performance liquid chromatography, with a valve body that has an inlet and at least two outlets that can be connected to the inlet in which a shutter element with shut-off surfaces can be used to alternately shut off the outlets directly by means of the conical or sphere segment shut-off surfaces.

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Samples separated by means of liquid separation, e.g. analytical or preparative liquid chromatography must be transported or collected in a suitable manner. Important criteria for the system and separating parameters are an optimum resolution per unit time, or a high flow rate of the substance to be isolated at a given purity. This is measured by means of suitable detectors. To separately collect the respective fractions, detector-controlled valves are used to divert the liquid flow leaving the analytical column into suitable receptacles.

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To this end, there are a series of prior-art valves that, however, all have certain disadvantages. A first decisive disadvantage is that a significant band widening can occur due to the valve construction, i.e., the desired separation does not occur. This can be due to the problematic channel path and design, and especially due to the poor design of the valve chambers, for example from dead zones such as blind holes or cavities that are poorly flushed, or by the internal valve volume. Some of the fraction can creep into these areas and mix with following fractions. In addition, substantial pressure peaks can sometimes arise when the valves are switched as the channels are briefly closed and/or when there are changes in volume when switching. These pressure peaks can lead to corresponding signal distortion in the chromatogram of the detectors used to control the valves, whereby an incorrect trigger pulse can be induced for the switching time of the valves. This can have a catastrophic effect on the quality of the fractions to be collected. Other disadvantages can arise in conjunction with the large construction-related forces that arise during switching and/or large forces that arise to ensure a sufficient seal when the valves are in the shut-off position. Large and strong drives are therefore necessary which run counter to the need for a very small installation space.

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Finally, depending on the respective valve constructions, the valves can unintentionally open due to the counterpressure on the outlet side, and essential parts that form the valve can be prematurely destroyed.

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A switch valve for liquid separation that has become known as a membrane valve and has the above-cited features consists of a valve body with an inlet channel that ends in a ring channel that is perpendicular to the inlet channel. The ring channel can be selectively sealed by one of two opposing shut-off cones whose rod-shaped extensions run in the centre of the ring channel, and the extensions contact each other for force transmis-

sion. The conical surfaces of the two shut-off cones oppose each other and are arranged so that they can seal the outlets of the ring channel designed as valve seats. The two shut-off cones are connected at the cone base to a membrane that is perpendicular to the ring channel and parallel to the inlet channel. These flexible membranes seal the liquid chambers connected to the outlets of the ring channel, whereby the liquid chambers connect with the actual outlet channels.

With this construction, there are particularly problematic blind-hole-like areas in the ring channel and in the liquid chambers assigned to the membrane. In these areas that have a poor or non-existent flow, the described remixing effects can occur with corresponding band widening.

With this double cone construction, liquid pressure that forms in the ring channel acts against the sealing force that is necessary to press one of the valve cones against the valve seat to seal one of the outlets. This requires correspondingly large holding and switching forces, hence this construction requires relatively large drive units.

In addition, a relatively large volume is displaced when switching the valves due to the membrane. This can make the valves sluggish. Furthermore, undesirable pressure peaks can arise that influence the detector signals so that the valves do not precisely switch which can produce undesirable band widening.

Another disadvantage of this construction is that membranes can be strongly stressed when pressure increases on the outlet side. This can lead to early membrane failure and, when the outlet channel is sealed, to the immediate destruction of the membranes.

It is therefore the problem of the invention to create a valve for liquid separation, especially for analytical or preparative liquid chromatography that avoids the disadvantages of the above-described state of the art.

This problem is solved by the features of patent claim 1, particularly in that the shut-off surfaces assigned to the outlet openings face away from each other.

By means of these relatively simple measures, a valve for liquid separation can be created where in particular the danger of band widening is reduced to a minimum by advantageous flushing. Another advantage of a valve designed in this manner is that the liquid pressure acting on the outlet side increases the sealing force, and the switching stroke is short with less volume displacement. In particular, small pulse drives can be used that enable frequent, precise and quick valve switching over a long time. When this valve is switched, undesirable pressure peaks do not arise that can affect the detectors via the inlet channel. By means of these measures, the purity of the collected fractions can be increased, and such valves are distinguished by a particularly long life.

The shut-off surfaces are usefully connected radially and symmetrically to an actuation axis of an actuator connected to the sealing element, preferably designed as a valve lifter. This favourably self-centres and hence positive seals the sealing element and produces a favourable bearing and seal of the actuator so that the essential valve parts operate reliably over a long time.

The sealing element is advantageously on the free end of the actuator. This produces a particularly favourable design of the valve body surrounding the sealing element and

accordingly of the liquid-conducting cavities in view of the particularly favourable rinsing behaviour that minimizes band widening.

Advantageously, the cross-section of the shut-off surfaces and the parts of the surface of the sealing elements opposite the inlet opening essentially forms a continuous line. The liquid flow is hence favourably guided and diverted without the formation of dead zones, and the sealing elements can be manufactured easily and precisely.

When one of the outlets is shut off, the sealing element with its shut-off surfaces assigned to this outlet advantageously lies on the opposing shut-off surfaces of a valve seat of the valve body and forms an annular sealing surface. This provides a good seal while the sealing element is centred on the valve seat, and it favourably guides the flow when open.

The opposing shut-off surfaces at the annular sealing surface advantageously form an angle with the actuation axis of the actuator that is greater or equal to 15° , and preferably greater than or equal to 30° . This advantageously centres the element without the danger of the sealing element locking to the valve seat.

The opposing shut-off surfaces of the valve seat usefully consist of a softer and elastic material, preferably Teflon, in contrast to the shut-off surface of the sealing elements. This produces particularly favourable friction and sealing conditions, and the valve parts are inert to the used liquids.

It is advantageous when the opposing shut-off surfaces at the sealing surface have a step or nose-like projection. This allows the shut-off surfaces of the sealing element to quickly reach a precise sealing position with a self-sealing effect under liquid pressure.

The opposing shut-off surface of the valve body that preferably faces the free end of the actuator narrows toward the outlet as a cone or funnel. This produces particularly good flushing behaviour of the internal hydraulic volume. This does a particularly good job of diverting the useful fraction into the primary channel.

The outlets on both sides of the inlet are advantageously opposite each other. This provides an advantageous seal with short flow paths without substantial pressure peaks when the valve is switched.

Both individually and together, the above measures contribute to the purity of the collected fractions without substantial band widening and without pressure peaks when the valve is switched; in addition, the drive units are small, the switching forces are low, and the valve has a long life.

Other features, aspects and advantages of the invention can be found in the following description with reference to the figures.

A preferred exemplary embodiment of the invention will be described in the following with reference to the figures. Shown are:

Fig. 1 a cross-section of a valve for liquid separation according to the invention;

Fig. 2 an enlarged cross-section of the valve body with the sealing element inside.

The valve 20 in Fig. 1 is designed as a three/two-way valve and has a valve body 21 with inlet 22 and a primary outlet 23 and secondary outlet 24. Inlet 22 has a connecting hole 67 to connect connecting lines (not shown) and ends in the cylindrical inlet channel 26. This ends in inlet 31 that is assigned to the switching chamber 35. The switching chamber 35 also has a primary outlet 32 and secondary outlet 33 that is opposite the primary outlet 32. The outlets 32, 33 are symmetrical to the actuation axis 51 and are on both sides of the inlet 31 at an angle of 90° to the inlet.

In the switching chamber 35 is first annular valve seat 36 assigned to the primary outlet 32 and the second annular valve seat 37 assigned to the second outlet 33. Also in the switching chamber 35 is the switchable sealing element 45 that can move in the direction of the actuation axis 51 and is rotatably mounted around the actuation axis 51, and the sealing element is between the first annular valve seat and the second annular valve seat. The spherical sealing element 45 is affixed to the actuator designed as a valve tappet 50 that is guided through the ring channel 55 connected to the second outlet 33 and has a cylindrical design. The valve tappet 50 is mounted and guided in the radial bearing 57, and its actuating end 63 has a fastener 64 that can be coupled to an actuating drive (not shown). This can especially be designed as a directional pulse generator or a spring-loaded retention magnet with an active and passive switching state, or a similar actuator.

The radial bearing 57 has holes 58, 59 through which the liquid in the ring channel 55 can pass. Contacting the radial bearing 57 is a radial seal 60 that has suitably designed ring lip zones so that it can assume a self-sealing function under liquid pressure.

The primary outlet 32 of the switching chamber 35 communicates with the primary outlet channel 27 of the ring flange 65 that has the connecting hole 66 of the primary outlet 23 to which suitable connecting lines can be connected. The connecting flange 65 can be screwed into and out of the threaded hole 69, and its sealing inner surface 77 perpendicular to the actuation axis 51 is on the contact surface 82 of the first annular valve seat 36. As can be seen in particular in Fig. 1, the entire valve 20 is easy to mount, adjust and remove. This allows the entire valve 20 with its essential valve parts to be quickly and easily cleaned.

The cylindrical switching chamber 35 has the inner diameter 42 and depth 43. The dimensions and volume of the switching chamber 35 are adapted to the valve parts inside it, that is, the first annular valve seat 36, the second annular valve seat 37, and the sealing element 45 attached to the free end 62 of the valve tappet 50 to yield a short switching stroke 75 of the sealing element 45 in connection with a low volume displacement. The liquid volume displaced when the valve 20 switches and the surface projection that determines the switching force are small enough to yield short switching times with small drives.

The first annular valve seat 36 has a cylindrical surface 81. It has an outer diameter that corresponds to the inner diameter 42 of the cylindrical switching chamber 35. In addition, the first annular valve seat 36 has a contact surface 82 that is perpendicular to the cylindrical surface 81. This continues radially outward to form the ring flange 38 of valve seat 36 that has an outer ring 39 on the outer edge. The first annular valve seat 36 is received by the contact ring 41 of the valve body 21 in a force fit and positive fit. The valve seat 36 also has a first opposing shut-off surface 84 angled inward into the

switching chamber 35 by angle 83 toward the actuation axis 51, and it extends over depth 85. The opposing shut-off surface 84 has a step or nose-shaped projection that forms the annular sealing lip 87.

- 5 The second cylindrical annular valve seat 37 is radially delimited by a cylindrical surface 91 whose outer diameter corresponds to the inner diameter 42 of the cylindrical switching chamber 35. The second annular valve seat 37 also has a contact surface 92 that is perpendicular to the cylindrical surface 91 that lies on the ring step 98 of the valve body 21. The second annular valve seat 37 also has a second opposing shut-off surface 94
10 formed in the angle 93 to the actuation axis 51 that extends the depth 95 of the second annular valve seat 37. The second opposing shut-off surface 94 has the step or nose-shaped projection 96 that forms the annular sealing lip 97.

- 15 The angle 83 of the first opposing shut-off surface 84 and the angle 93 of the second opposing shut-off surface 94 are approximately 30 degrees in the exemplary embodiment and provides a favourable, centred and slightly sealing contact for the sealing elements 45 without the sealing element 45 locking at the annular sealing surface 78 while the first and second annular valve seats 36, 37 are sealed.

- 20 The inner diameter of the first annular valve seat 36 transitions into the conically narrowing opening surface 34 of the primary outlet 32, i.e., toward the actuation axis 51. This forms an outlet area 40 with a particularly favourable flow that is delimited by the first opposing shut-off surface 84 and the annular opening surface 34. The inner diameter of the second annular valve seat 37 is the same size or slightly bigger than the diameter 54 of hole 53 so that the design of this area is also favourable to flow.
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- The sealing element 45 between the two valve seats 36 and 37 is a spherical segment with a radius 73 and has a regular cylindrical hole 46. When uninstalled, its inner diameter 47 is slightly smaller than the outer diameter 52 of the cylindrical valve tappet 50 so that a tight press seat of the sealing element 45 on the valve tappet 50 results when installed. The sealing element 45 has a first shut-off surface 48 that faces a first opposing shut-off surface 84, and a second shut-off surface 49 facing the second opposing shut-off surface 94. Due to the spherical shape of sealing element 45, the first shut-off surface 48 and second shut-off surface 49 are designed to face away from each other.
30 The first shut-off surface 48 and second shut-off surface 49 form a part of the spherical or circular section 71 that is arced with radius 73. Hence the sealing element 45, is designed in an essentially continuous line 72 between the inlet 31 and the first and second opposing shut-off surfaces 84, 94.
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- 40 The sealing element 45 consists of high-grade steel while the first and the second annular valve seats 36 and 37 consist of softer, elastic Teflon. The contact and seal are therefore favourable, and the Teflon is inert to the liquids of the collected fractions.

- 45 The functioning of the valve 20 will be further described below:

- In the position shown in Fig. 1 and 2, the sealing element 45 with its second shut-off surface 49 seals against the second opposing shut-off surface 94 of the second annular valve seat 37 and also seals the secondary outlet 33. In this position, a narrow annular gap 70 is formed between the first shut-off surface 48 of sealing element 45 and the first opposing shut-off surface 84 of the first annular valve seat 36 through which an optimised flow of liquid passes following the arrow 25 through the inlet channel 26 and inlet
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31 into the switching chamber 35 of the valve body 21 to fill the part of the switching chamber 35 assigned to the primary outlet 32 and the outlet chamber 40. The liquid can leave the primary outlet chamber 40 in the direction of the arrow 29. In this manner, the liquid can be collected in a suitable receptacle during a first interval assigned to the first fraction, preferably the useful fraction of the liquid.

As soon as the drive unit (controlled by a upstream detector (not shown) before the valve 20) rapidly switches the valve 20, the valve tappet 50 bearing the sealing element 45 is shifted transversely in the direction of the arrow 29. The sealing element 45 moves in the direction of the arrow 29 until its first shut-off surface 48 seals the step or nose-shaped projection 86 of the first opposing shut-off surface 84 designed as a sealing lip 87. In the brief transition period in which the first shut-off surface 48 has not yet reached the projection 86 of the first opposing shut-off surface 84, neither the primary outlet 32 nor the secondary outlet 33 are sealed by the sealing element 45. This prevents the formation of pressure peaks.

When the sealing element 45 contacts the sealing lip 87, the pressurized liquid entering the inlet channel 26 and inlet 31 into the switching chamber 35 and leaving through ring channel 55 independently and additionally presses the first shut-off surface 48 of the sealing elements 45 against the first opposing shut-off surface 84 of the first annular valve seat 36 so that the forming liquid pressure effectively supports the sealing force. The projection 86 shaped as a sealing lip 87 of the first opposing shut-off surface 84 deforms slightly so that there is a sufficiently wide annular sealing surface available to form a seal.

In this switched state (not shown in Fig. 1 and 2) in which the sealing element 45 is pressed against the first annular valve seat 36, the liquid of the second fraction entering in the direction of the arrow 25, preferably consisting of the eluents, passes through the ring channel 55. The arrangement and design of the inlet channel 26 of the sealing element 45, second annular valve seat 37 and switching chamber 35 produces a swirling flow of liquid. This causes the liquid to advantageously and completely circulate around the valve tappet 50 and through the ring channel 55. The second fraction can then flow through the second outlet channel 28 (arrow 30) and is available at the second outlet 24 with the connecting hole 68 to be collected in a suitable receptacle.

Numerous additional switching operations can follow corresponding to the described steps.

In the exemplary embodiment in Fig. 1 and 2, valve 20 has an asymmetrical liquid path that is designed for optimum flow and circulation (arrows 25 and 29). This is also attained by placing inlet 31 directly next to the second annular valve seat 37 so that the narrow sealing gap between the second shut-off surface 49 of sealing element 45 and the second opposing shut-off surface 49 of the second annular valve seat 37 are optimally flushed. This effect can be increased by angling or tilting the inlet channel 26 so that the direction of the flow induced through the channel 26 forms an acute angle with the actuation axis 51. The flushing and outflowing effect can be further enhanced by angling the inlet channel 26 eccentric to the actuation axis 51 so that the flow formed through the inlet channel 26 runs at a distance from the actuation axis 51 so that the liquid flow through the inlet 31 into the switching chamber 35 swirls around the actuation axis 51.

Of course, beyond the free end 62 of the valve tappet 50, the sealing element 45 can also be located in the area of a part of the valve tappet 50 shifted toward the actuating end 51 so that a ring channel corresponding to ring channel 55 can be formed to either side of the sealing elements 45. In addition, there can also be several sealing elements 45 affixed to the single valve tappet 50 that are offset axially toward the actuation axis 51, and several additional inlet and outlet channels can be correspondingly created. This allows several liquid-conducting channels to be advantageously switched simultaneously.

I CLAIM:

5 1. A valve for liquid separation, especially for analytical or preparative liquid chromatography, comprising a valve body having an inlet and at least two outlets, a sealing element coupled with the inlet and outlet, the sealing element including shut-off surfaces for alternately shutting off the outlets, the shut-off surfaces including a segment shaped as a segment of one of a cone and sphere, the shut-off surfaces respectively associated with the outlets being arranged to face away from each other.

2. The valve of claim 1 wherein the shut-off surfaces are radially symmetrical to an actuation axis of an actuator for the sealing element.

3. The valve of claim 2 wherein the actuator includes a valve tappet connected to the sealing element.

4. The valve of claim 2 wherein the sealing element is at a free end of the actuator.

5. The valve of claim 3 wherein a cross-section of the shut-off surfaces and surface parts of the sealing element opposite the inlet form an essentially continuous line.

6. The valve of claim 1 wherein the outlets and sealing element are arranged so that when one of the outlets is shut off, the sealing element with its shut-off surface associated with its respective outlet rests on opposing shut-off surfaces of a valve seat of the valve body to form an annular sealing surface.

7. The valve of claim 5 wherein the opposing shut-off surfaces form an angle having an actuation axis at the annular sealing surface that is greater than or equal to 15°.

8. The valve of claim 7 wherein the angle is at least 30°.

9. The valve of claim 6 wherein the opposing shut-off surface of the valve seat includes material that is softer and more elastic than the shut-off surfaces of the sealing

elements.

10. The valve of claim 9 wherein the valve seat material is TEFLON.

11. The valve of claim 6 wherein the opposing shut-off surfaces have a step or nose-shaped projection at the annular sealing surfaces.

12. The valve of claim 6 wherein the opposing shut-off surface of the valve body narrows conically or as a funnel toward at least one of the outlets.

13. The valve of claim 12 wherein the opposing shut-off surface opposes the free end of the actuator.

14. The valve of claim 1 wherein the outlets on both sides of the inlet oppose each other.

ABSTRACT

A valve for liquid separation, especially for analytical or preparative liquid chromatography, includes a valve body having an inlet and at least two outlets connected to the inlet for a flow of liquid. The valve body includes a sealing element having shut-off surfaces for alternately shutting off the outlets. The shut-off surfaces face away from each other and have the shape of a cone segment or spherical segment.

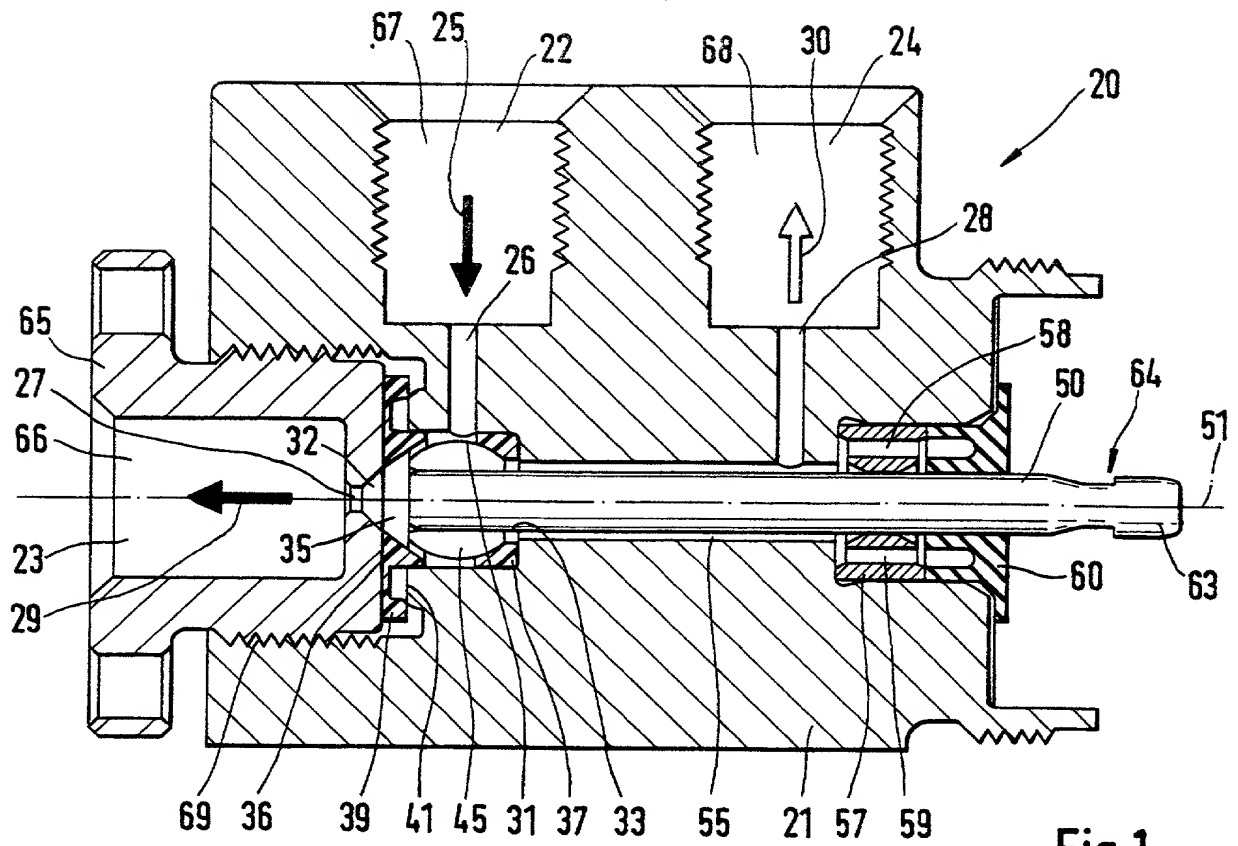


Fig.1

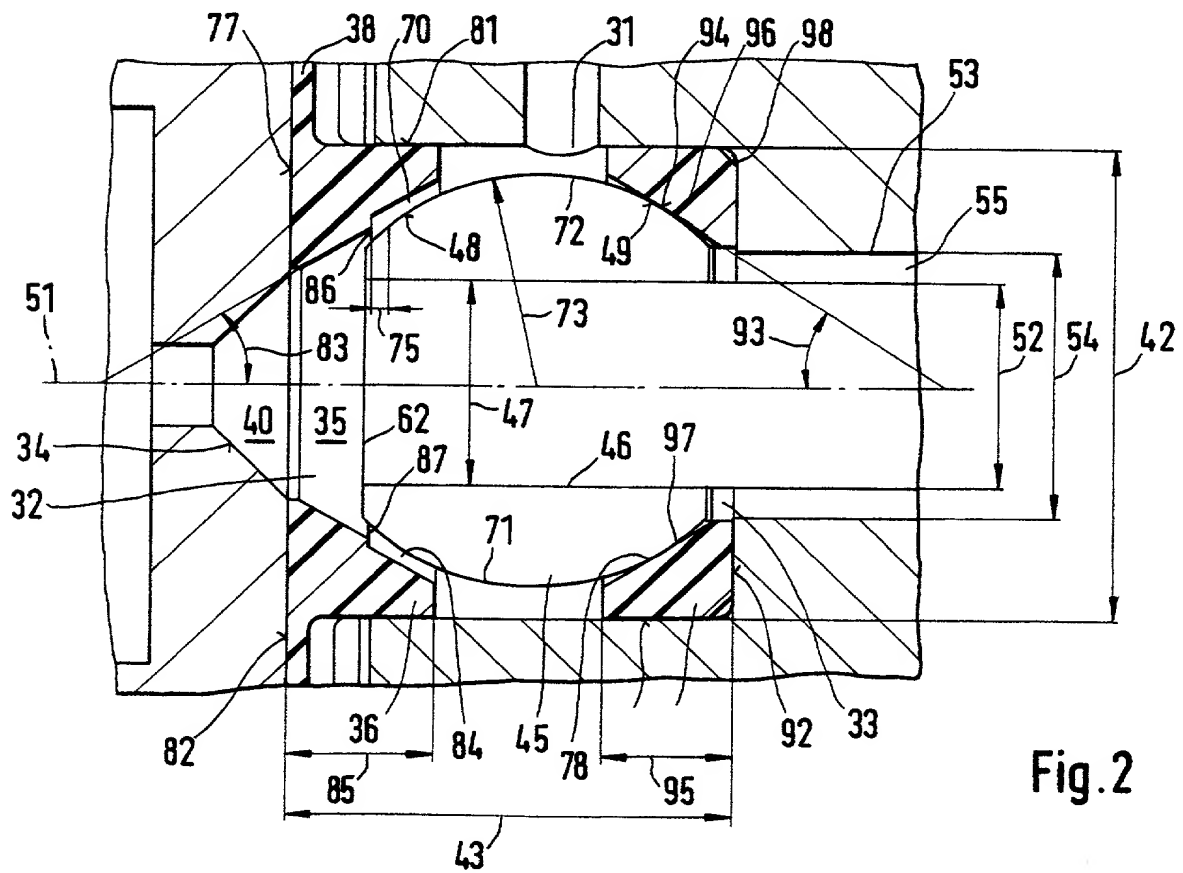


Fig.2